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(19)

(54) SEPARATION OF OLEFIN POLYMERS

(71) We, NATIONAL PETRO-CHEMICALS CORPORATION, a corporation organised and existing under the laws of the State of Delaware, United States of America, of 99 Park Avenue, City and State of New York, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to the separation of an olefin polymer from a slurry thereof in a liquid diluent.

In the polymerization of olefins in accordance with the particle form polymerization process, the effluent from the polymerization reactor is in the form of a slurry of the solid olefin polymer in a diluent. Many methods have previously been proposed for removal of the product slurry from the polymerization reactor and subsequent separation of the liquid diluent from the solid polymer.

It is known to carry out the particle form polymerization of ethylene, for example, in a continuous loop reactor from which the product slurry may be withdrawn through a settling leg or legs which are periodically discharged. The use of such a system poses certain problems in view of the relative mechanical complexity of the settling legs and the undesirable pressure pulses which are created in the loop reactor by the periodic discharge of the settling legs.

In accordance with the present invention a process for the polymerization of an olefin comprises producing a slurry of the solid olefin polymer in a liquid diluent within a polymerization zone, passing the product slurry directly to a separation zone, removing from the separation zone an overflow stream concentrated in the liquid diluent, and an underflow stream concentrated in the solid polymer, taking off a portion of the underflow stream for recovery of the solid polymer, combining the overflow stream with the residual portion of the underflow stream, and recycling the resulting mixture to the polymerization zone. By thus providing a by-pass arrangement in a separation zone, e.g. in a cyclone separator, smooth and continual flow of the product slurry from the polymerization zone and through the separation zone is obtainable.

In accordance with a preferred feature of the invention, a pressure gradient is maintained in the polymerization zone between the region from which the polymer slurry is removed and that to which the recycle mixture is returned, the pressure gradient (e.g. of from 5 to 10 psi) providing a motive force for the feed to the separation zone and the recycle therefrom.

The use of a cyclone separator in the separation of solid olefin polymer from a polymerization reactor effluent slurry is disclosed in U.S. Patent No. 3,418,305. This patent does not, however, suggest bypassing the overflow from such a separator and recycling the same together with a portion of the separator underflow to avoid blockage and maintain a constant volumetric flow through the separation system. Nor does it suggest connecting the cyclone separator across the polymerization reactor in such a manner as to create a pressure gradient between the take-off from and recycle to the reactor, which gradient motivates the fluid stream through the separation system. It is these features, together with elimination of the use of the settling legs commonly employed for removing olefin polymer product from slurries produced in the particle form polymerization process, which characterize the preferred embodiments of the present invention.

The process of the present invention may be employed in the separation of any olefin polymer from a slurry containing the same in a suitable liquid diluent as formed in the well known particle form technique for the polymerization of olefin monomers. The present separation technique is particularly useful in conjunction with olefin polymerizations carried out in continuous loop reactors such as described, for example, in U.S. Patent No. 3,248,179. It may also be used in cooperation with any of the various polymer drying or diluent separation and recovery or recycling operations disclosed, for example, in U.S. Patent No. 3,152,872, U.S. Patent No. 3,318,857, or the aforesaid U.S. Patent No. 3,418,305.

The present invention is useful in the separation and recovery of polymers of any suitable α-olefins formed in accordance with the known low pressure polymerization techniques carried out in the presence of chromium oxide, organometallic or other catalyst systems. Preferably, the process is utilized in the separation of ethylene polymers, e.g. those formed over organometallic catalysts. However, it may be used in connection with the polymerization of any of the monomers, employing any of the catalysts, or in the presence of the diluents, modifiers or the like, and utilizing the known reaction temperatures, pressures and other parameters, which are commonly used in the art. Such compositions and process conditions with which the separation process of this invention may be used are described, for example, in the above-noted patents which, it will be understood, are cited as illustrative only.

In a particularly preferred form of the invention, illustrated in the attached drawing, a loop reactor 1 is illustrated into which an olefin monomer stream 2 and a diluent stream 3 are fed through a line 4. A catalyst, suitably introduced as a suspension in the diluent, is also introduced into the loop reactor 1 through a separate line 5. The reaction mixture is continuously circulated through the reactor, in the direction designated by arrows 6, by an impeller 7 connected through an appropriate seal and driven by a motor 8. The temperature and pressure conditions within the loop reactor are suitably controlled so as to effect polymerization of the olefin monomer continuously circulated therethrough and as to form a slurry of the desired solid olefin polymer within the reactor.

The polymer slurry is removed from the loop reactor 1 through a valved (9) line 10 and into and through a cyclone separator 11. The separator acts as a liquid cyclone thickener, the relatively large solid polymer particles tending to be separated as an underflow stream and the liquid diluent together with any residual monomer or other fluids and fines tending to separate as an overflow stream. The solid polymer-enriched underflow is removed as stream 12, a portion of which is taken off through valved (13) line 14 for recovery and the residual portion of which is fed through line 15 for recycle. The overflow stream, on the other hand, is removed from the separator through a by-pass line 16, combined with the residual underflow in line 15, and recycled to reactor 1 through a valved (17) line 18. It is this by-pass line which, as indicated hereinabove, insures a constant volumetric flow through the cyclone separator and prevents blockage thereof by the high solids content slurries fed therethrough.

The product slurry removed through line 14 is enriched in the desired solid polymer product. For example, polyethylene-containing slurries having solids contents of from 40% to 65% by weight may be thus separated and removed for subsequent drying and recovery. By utilizing the separation technique hereof, it is possible to decrease subsequent after-treatment while simultaneously effecting at least a partial classification of the desired polymer. The concentrated polymer product is then conventionally treated, e.g., in a flash hopper 19, the solid polymer being recovered through line 20 and vaporized diluent being removed through line 21 (see, for example, the aforesaid U.S. Patent No. 3,152,872).

It should be further noted that the slurry take-off line 10 and the recycle line 18 are connected to loop reactor 1 downstream and upstream, respectively, of the impeller 7 for the reaction mixture. The pressure drop across this pump provides a pressure gradient serving to motivate fluid flow through line 10 into the cyclone separator 11, through the separation system, and through return or recycle line 18. Employing an axial impeller operating at 20,000 G.P.M. at 9.3 psi pressure, gradients or heads of from about 5 to 10 psi may thus be established.

In the manner described hereinabove, polymer slurries removed from the loop reactor 1 through line 10 having solids contents of about 25 weight percent may be readily concentrated to product slurries recovered through line 14 having solids contents of up to 65 weight percent. Yet such is accomplished, as indicated hereinabove, without the use of settling legs, without blockage problems, and without the need for external pumping means (the pressure differentials within the loop reactor itself motivating the separation).

WHAT WE CLAIM IS:—

1. A process for the polymerisation of an olefin which comprises producing a slurry of the solid olefin polymer in a liquid diluent within a polymerisation zone, passing the product slurry directly to a separation zone, removing from the separation zone an overflow stream concentrated in the liquid diluent and an underflow stream concentrated in the solid polymer, taking off a portion of the underflow stream for recovery of the solid polymer, combining the overflow stream with the residual portion of the underflow stream, and recycling the resulting mixture to the polymerisation zone.

2. A process according to claim 1 wherein a pressure gradient is maintained in the polymerisation zone between the region from which the polymer slurry is removed and that to which the recycle mixture is returned, the pressure gradient providing a motive force for the feed to the separation zone and the recycle therefrom.

3. A process according to claim 2 wherein the pressure gradient is from 5 to 10 psi.

4. A process according to claim 1, 2 or 3 wherein the separation zone comprises a cyclone separator.

5. A process according to any of claims 1 to 4 wherein the production of the product slurry of solid olefin polymer is conducted in a continuous loop reaction zone.

6. A process according to any of claims 1 to 5, for the polymerisation of ethylene, in which the said taken off portion of the underflow stream has a solids content of from 40% to 65% by weight.

7. A process according to claim 1 for the polymerisation of an olefin substantially as hereinbefore described with reference to the accompanying drawings.

8. An olefin polymer obtained by a process according to any one of claims 1 to 7.

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1 SHEET This drawing is a reproduction of the Original on a reduced scale